

## CROSS SECTIONING OF OPTOELECTRONIC DEVICE

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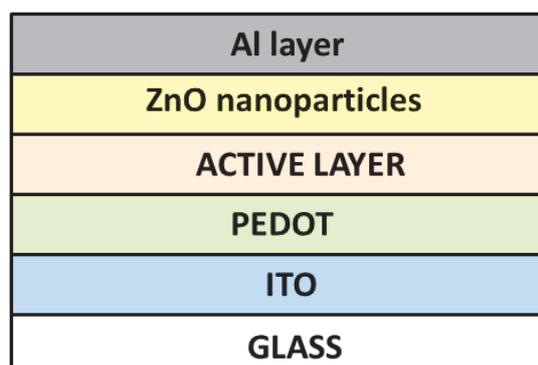
### Abstract

Optoelectronic devices play very important role in life nowadays. Most of the devices are widely used in fields ranging from image processing and fiber optic communication to common consumer electronics (twilight switches, house security systems, etc.), where components such as photodiodes, laser diodes, phototransistors, photomultipliers, optical isolators, LEDs or OLEDs are mounted. Accurate procedure is necessary during the preparation to achieve their fully functionality. Because the devices are mostly prepared as multilayer systems, there is a requirement for correct functional thickness of layers. The thickness of the layers could be checked not only during the deposition but also retrospectively in the case of some failures during their deposition. The method of ion beam milling can be used for cross section preparation and can achieve cross sections of soft materials or material combinations consisting of hard and soft components, which are used in devices. This contribution deals with case study of SEM thickness layer characterization of optoelectronic device on cross section, which was prepared by ion milling.

**Keywords:** Ion beam milling, optoelectronic device, scanning electron microscopy (SEM), diamond saw

### 1. INTRODUCTION

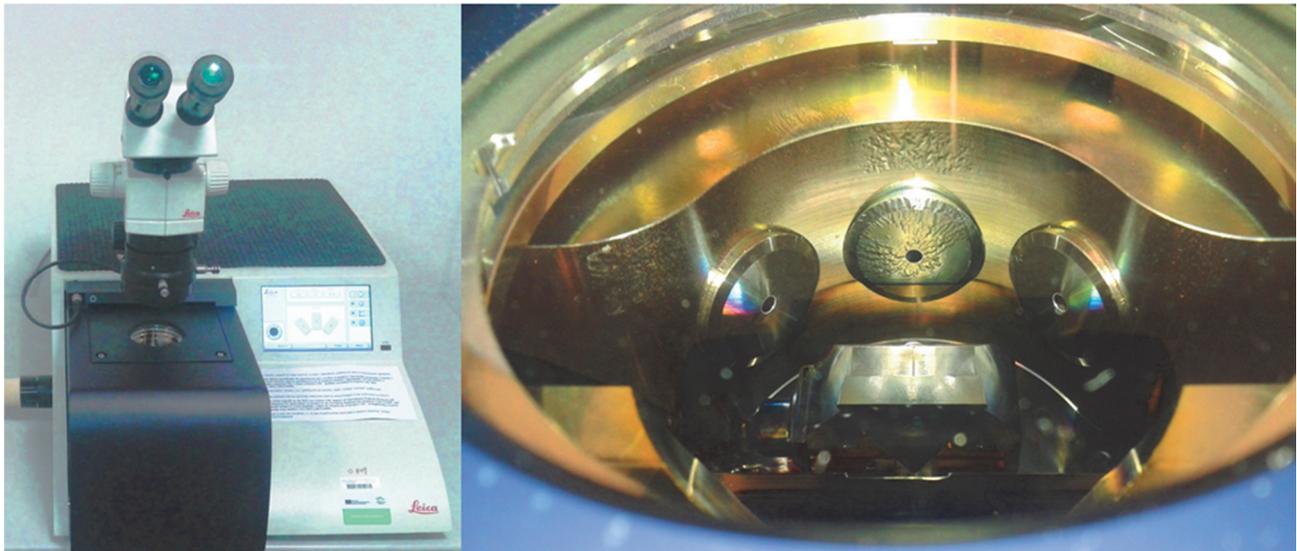
Optoelectronic devices play very important role in life nowadays. Optoelectronics elements are classified into different types such as light emitting diode (LED), photodiode, solar cells, optical fiber, laser diodes, etc. [1]. Highly used element is first named LED, the device generating light when suitable voltage is applied. The using of organic films and foils' substrates films in optoelectronics allow to produce flexible elements nowadays. Because the devices are mostly prepared as multilayer systems, there is a requirement for correct functional thickness of layers. The thickness of the layers could be checked not only during the deposition but also retrospectively in the case of some failures during their deposition. The method of ion beam milling can be used for cross section preparation and can achieve cross sections of soft materials or material combinations consisting of hard and soft components [2], which are used in devices.



**Figure 1** Construction of LED

## 2. ION BEAM MILLING

Ion beam is a powerful tool for a various application and systems operating focused ion beam have been produced for over twenty years. Nowadays the application in dual beam systems (combination of SEM and focused ion beam - FIB) is very popular, which is used for patterning or modifying of surface in nanoscale and then the patterned surface is examined by SEM. Focused ion beam can be used for lithography or FIB imaging as well. Ion beam plays also very important role in sample preparation for electron microscopy and also in cross sectioning of hard/soft, porous, heat sensitive, brittle and heterogeneous material for (SEM), microstructure analysis (EDS, WDS, Auger, EBSD) and, AFM investigations (**Figure 2**) [3].



**Figure 2** Ion beam milling system Leica EM TIC 3X (right) and the detail of chamber with 3 ion guns (left)

## 3. SAMPLE PREPARATION

### 3.1. Cutting and polishing

Sample LED diode (**Figure 1**) was on glass substrate and before ion beam milling was necessary to cut it in the middle of diode, where all active layers were deposited (sandwich structure). The cutting was done by diamond saw with 15 000 rpm cooled with water. After cutting, the sample was polished by sand paper with roughness of 2  $\mu\text{m}$  and subsequently 0.9  $\mu\text{m}$  at 2500 rpm with water cooling. All this procedures were carried out by instrument Leica EM TXP.

### 3.2. Ion beam milling

Thereafter the sample diode was milled by ion beam in chamber of Leica TIC 3X instrument (**Figure 2**). Because the diode consisting of hard and soft material combination it was necessary to use sample cooling by liquid nitrogen to avoid melting of polymer layers during the ion bombardment. Argon is used as the process gas. The chamber was evacuated to  $9 \cdot 10^{-5}$  mbar and working pressure in the chamber was set to  $2 \cdot 10^{-4}$  mbar.

The process was consisting of 3 steps:

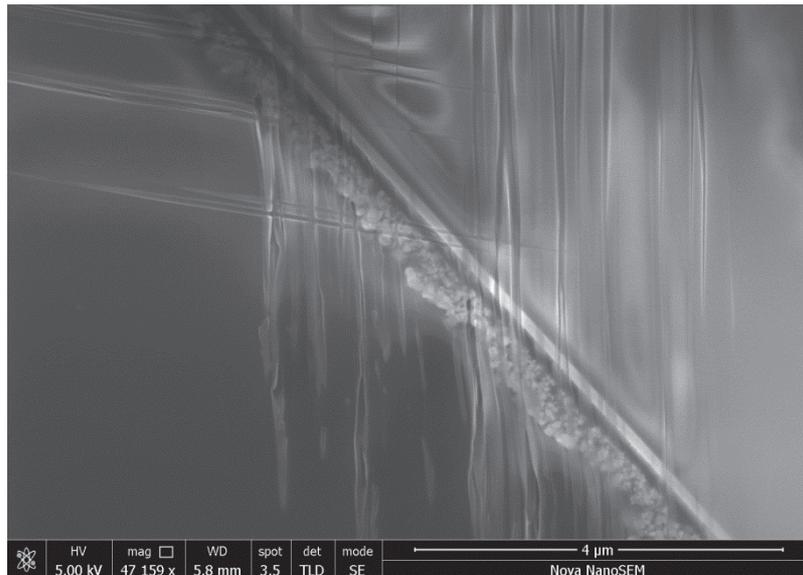
- 1) Time of process 3 hours, ion guns accelerating voltage of 6 kV, current of 2.2 mA, the sample temperature: 0 °C (the temperature of the sample could be higher because the cover glass was etched by ion beam first)
- 2) Time of process 3 hours, ion guns accelerating voltage of 6 kV, current of 2.2 mA, the sample temperature: -15 °C)

- 3) Time of process 3 hours, ion guns accelerating voltage of 6 kV, current of 2.2 mA, the sample temperature: -15 °C)

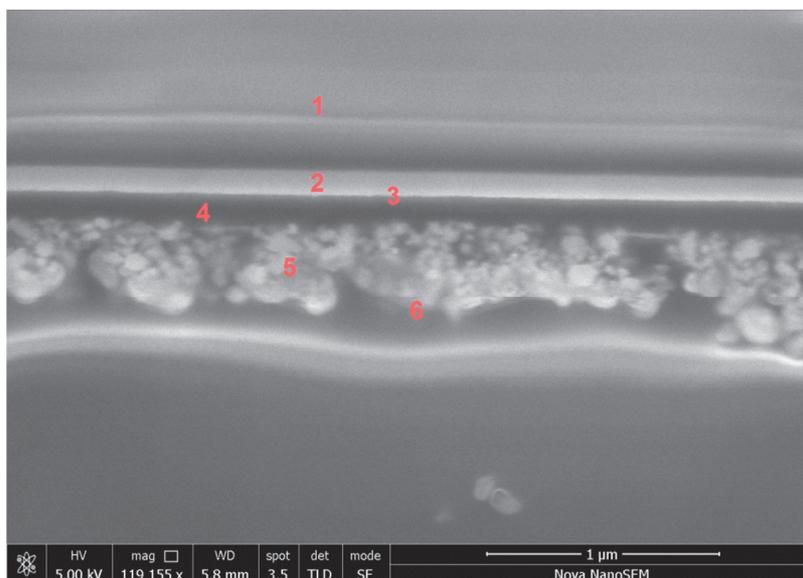
After these steps the sample diode was etched through all layers and was prepared for SEM inspection.

#### 4. RESULTS

The processed sample diode was inspected by scanning electron microscope FEI Nova NanoSEM 450 and the thickness of individual layers were determined (**Figure 3** and **Figure 4**).



**Figure 3** Individual layers of LED diode with visible traces after ion beam milling



**Figure 4** Individual layers of LED diode (1-substrate glass, 2 - ITO, 3 - PEDOT, 4 - active layers [4], 5 - ZnO nanoparticles, 6 - sputtered layer of aluminum [5])

The thickness of individual layers are following; ITO layer of 109 nm, PEDOT layer of 47 nm, active layer of 80 nm and layer of ZnO nanoparticles varies from 150 to 390 nm because the layer is not homogenous and nanoparticles differ in their size.

## 5. CONCLUSION

We successfully prepared cross section of LED diode by ion milling system. This cross section of hard and soft material combination was prepared by ion milling with liquid nitrogen cooled sample. We determined the thickness of layers composing LED diode prepared by ourselves, so we are able to control layer thickness not only during the deposition but also retrospectively.

## ACKNOWLEDGEMENTS

*This article was written with the support of Operational Program Research and Development for Innovations co-funded by the European Regional Development Fund (ERDF) and national budget of the Czech Republic, within the framework of the project CPS - strengthening research capacity (reg. number: CZ.1.05/2.1.00/19.0409).*

*This work was also supported by the Ministry of Education, Youth and Sports of the Czech Republic - program NPU I (LO1504).*

## REFERENCES

- [1] HU, W. *Organic optoelectronic*, WILEY-VCH Verlag GmbH & Co. KGaA, Weinheim, 526 p., 2013, ISBN 978-3-527-32968-7.
- [2] GRUNEWALD, W. Cross Sectioning of Rubber (Tire), *Application Note for Leica EM TIC 3X - Industrial Manufacturing*, [quoted 2016-09-30]., 2016, <https://www.leica-microsystems.com/science-lab/cross-sectioning-of-rubber-tire/>
- [3] Leica Microsystem GmbH (DE), Ion Beam Milling System Leica EM TIC 3X, [quoted 2016-09-30]., 2016, <https://www.leica-microsystems.com/products/sample-preparation-for-electron-microscopy/ion-beam-milling-systems/details/product/leica-em-tic-3x/>
- [4] KRCMAR, P. et al. Active layer for electroluminescent foils, comprises electrically conducting polymer based on 2-methoxy- and 5-(2-ethylhexyloxy) substituted poly-1,4-phenylenevinylene and inorganic filler based on zinc oxide nanoparticles, Patent Number(s): CZ201200916-A3; CZ304387-B6.
- [5] KOLARIK, V. et al., Institute of Scientific Instrument: An Overview Presentation, *In Proc's of METAL 2012, 21th Int'l Conference on Metallurgy and Materials*. May 23-25, 2012, Brno, TANGER Ltd, Ostrava, 2012, p. 26-31, ISBN 978-80-87294-31-4.